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[INVENTOR]
[Address or Residence] c/o SHISEIDO RESEARCH CENTER(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa
[Name] Tetsuya Kanemaru

[INVENTOR]
[Address or Residence] c/o SHISEIDO RESEARCH CENTER(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa
[Name] Kyoko Jouichi

[INVENTOR]
[Address or Residence] c/o SHISEIDO RESEARCH CENTER(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa
[Name] Kazuhisa Ohno

[APPLICANT]
[Identification Number] 000001959
[Name of Applicant] SHISEIDO COMPANY, LTD.

[PATENT ATTORNEY]
[Identification Number] 100090527
[Name of Patent Attorney] Chieko Tateno
[Phone Number] 03-5731-9081

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[NAME OF DOCUMENT] Specification

[TITLE OF THE INVENTION] Silicone-Treated Powder, Process of Production Thereof, and Composition Using the Same

[SCOPE OF CLAIM FOR PATENT]

[Claim 1] A silicone-treated powder comprised of a powder coated on its surface with a silicone compound, said silicone-treated powder characterized in that an amount of hydrogen produced by Si-H groups residually present on the surface of the silicone-treated powder is not more than 0.2 ml/g of treated powder and a contact angle of water with the treated powder is at least 100°.

[Claim 2] A cosmetic composition characterized by using the silicone-treated powder as claimed in claim 1 as one ingredient of the material.

[Claim 3] A cosmetic composition as claimed in claim 2, wherein said cosmetic composition is one of a solid foundation, emulsion foundation, pressed powder, face powder, UV blocking stick, lipstick, water-in-oil type emulsion sunscreen, and body powder.

[Claim 4] A paint characterized by using the silicone-treated powder as claimed in claim 1 as one ingredient of the material.

[Claim 5] A resin shaped article characterized by being obtained by injection molding a synthetic resin composition using the silicone-treated powder as claimed in claim 1 as one ingredient of the material.

[Claim 6] A process of production of a silicone-treated powder characterized by comprising

a first step of coating a surface of a powder with

(1) a silicone compound having at least one Si-H group or

(2) a mixture of the silicone composition of (1) and a silicone composition not having an Si-H group and

a second step of heating the silicone compound coated powder at 350 to 500°C for 0.1 to 24 hours.

[Claim 7] A process of production of a silicone-treated powder as claimed in claim 6, wherein said silicone compound

having an Si-H group is a silicone compound of the following general formula (1)

[Chemical 1]

$(R^1HSiO)_a(R^2R^3SiO)_b(R^4R^5R^6SiO_{1/2})_c$ ... (1)

[wherein, (R^1, R^2, and R^3 are mutually independently a hydrogen atom or a C_1 to C_{10} hydrocarbon group substitutable by at least one halogen atom (provided that R^1, R^2, and R^3 are never simultaneously hydrogen atoms), R^4, R^5, and R^6 are mutually independently a hydrogen atom or a C_1 to C_{10} hydrocarbon group substitutable by at least one halogen atom, a is an integer of 1 or more, b is 0 or an integer of 1 or more, c is 0 or 2 (provided that $3 \leq a+b+c \leq 10000$), and the compound includes at least one Si-H group portion].

[Claim 8] A process of production of a silicone-treated powder as claimed in claim 7, wherein said silicone compound having an Si-H group is methylhydrogenpolysiloxane, a methylhydrogenpolysiloxane-dimethylpolysiloxane copolymer, or tetramethylcyclotetrasiloxane.

[Claim 9] A process of production of a silicone-treated powder as claimed in claim 6, wherein said heat treatment in said second step is performed in the air or under an atmosphere of one or more other types of gases containing moisture of at least an extent of the moisture in the air or under an atmosphere not containing moisture while adding moisture.

[Claim 10] A cosmetic composition using a silicone-treated powder obtained by the process of production as claimed in claim 6 as one ingredient of the material.

[Claim 11] A cosmetic composition as claimed in claim 12, wherein said cosmetic composition is one of a solid foundation, emulsion foundation, pressed powder, face powder, UV blocking stick, lipstick, water-in-oil type emulsion sunscreen, and body powder.

[Claim 12] A paint characterized by using a silicone-treated powder obtained by the process of production as claimed in claim 6 as one ingredient of the material.

[Claim 13] A resin shaped article characterized by being obtained by injection molding a synthetic resin composition using a silicone-treated powder obtained by the process of production as claimed in claim 6 as one ingredient of the material.

[DETAILED DESCRIPTION OF THE INVENTION]

[0001]

[Technical Field of the Invention]

The present invention relates to a process of production of a silicone-treated powder, more particularly relates to a silicone-treated powder obtained by coating a silicone compound having an Si-H group on the surface of a powder and polymerizing the silicone on the surface of the powder by heat treatment to bring out water repellency and eliminate almost all the residual Si-H groups on the coating, able to be blended into various cosmetics, and superior in stability in a product, and a process of production of the same.

[0002]

[Prior Art]

There have been various methods for giving hydrophobicity to a powder in the past. Using the hydrophobicity of silicone oil is well known.

The silicone compound usable for giving hydrophobicity means one having an organohydrogenpolysiloxane chain in the molecule and sometimes also having a diorganopolysiloxane chain in the molecule or a mixture of organohydrogenpolysiloxane and diorganopolysiloxane. When these are coated on the surface of a powder, due to the surface activity of the powder, the Si-H group bonded portion of the organohydrogenpolysiloxane molecule reacts with the moisture etc. in the air, the Si-OH groups produced react with the Si-H groups of the adjoining other molecules, or the Si-OH groups react among themselves to cause cross-linking and polymerization and form a silicone film.

With heat treatment in the air at about 200°C after coating organohydrogenpolysiloxane on the surface of a powder,

however, while the cross-linking reaction of the molecules themselves proceeds to a certain extent, the residual Si-H groups are not completely eliminated. On the other hand, with heating at over 500°C, the silicone starts to burn and is converted to silica (Japanese Unexamined Patent Publication (Kokai) No. 11-199458, heating at 600 to 950°C for treatment for coating silicon oxide).

Such residual Si-H groups react with the moisture in the air or the moisture, alcohol, amines, etc. in makeup products over a long period to cause the production of hydrogen and form new siloxane bonds, so if the above treated powder is used as it is for cosmetics, paints, toners, inks, containers, and ingredients of various other compositions, various problems will sometimes be caused in the compositions.

For example, with cosmetics, there is the risk of production of hydrogen in the process of production, the containers may swell after the elapse of time after filling the product into containers, and the product may harden and crack. In the case of paints, the problem of deterioration of the container sometimes occurs.

[0003]

[Problems to be Solved by the Invention]

To reduce the above residual Si-H groups, for example, the method of Japanese Unexamined Patent Publication (Kokai) No. 63-113081 (Japanese Patent No. 1635593) (addition of compound having unsaturated hydrocarbon group to residual Si-H groups by hydrosilylation reaction), the method of Japanese Unexamined Patent Publication (Kokai) No. 8-192101 (substitution of residual Si-H groups by contact with water or lower alcohol), the method of Japanese Examined Patent Publication (Kokoku) No. 56-43264 (mixture and pulverization of metal hydroxide serving as catalyst for cross-linking and polymerization of organohydrogenpolysiloxane with treated powder, then using mechanochemical reaction), etc. have been attempted.

The above methods are effective in their own right, but

the processes are complicated, a long time is required, or relatively active functional groups are adsorbed on the surface, so the powder is given an unpleasant smell etc.

[0004]

The present invention was made in view of the above situation and has as its object the provision of a silicone-treated powder free from production of hydrogen and of a good quality and also a process for production of such a silicone-treated powder and a process of production reduced in manufacturing cost.

[0005]

[Means for Solving the Problems]

The present inventors engaged in intensive research and as a result discovered that by heating a powder coated with organohydrogenpolysiloxane etc. at 350 to 500°C, it is possible to cross-link or substitute with inert functional groups almost all of the residual Si-H groups while maintaining the hydrophobicity and thereby completed the present invention.

[0006]

That is, the present invention provides a silicone-treated powder comprised of a powder coated on its surface with a silicone compound, said silicone-treated powder characterized in that an amount of hydrogen produced by Si-H groups residually present on the surface of the silicone-treated powder is not more than 0.2 ml/g of treated powder and a contact angle of water with the treated powder is at least 100°.

[0007]

Further, the process of production of a silicone-treated powder according to the present invention is characterized by comprising

a first step of coating a surface of a powder with

(1) a silicone compound having at least one Si-H group or

(2) a mixture of the silicone composition of (1) and a silicone composition not having an Si-H group and

a second step of heating the silicone compound coated powder at 350 to 500°C for 0.1 to 24 hours.

[0008]

Further, according to the present invention, there are provided a cosmetic composition characterized by using the above silicone-treated powder as one ingredient of the material, a paint characterized by using the above silicone-treated powder as one ingredient of the material, and a resin shaped article characterized by being obtained by injection molding a synthetic resin composition using the silicone-treated powder as one ingredient of the material. Here, as the cosmetic composition, any of a solid foundation, emulsion foundation, pressed powder, face powder, UV blocking stick, lipstick, water-in-oil type emulsion sunscreen, and body powder is preferable.

[0009]

[Mode of Carrying out the Invention]

The present invention will be explained in more detail below.

The powder usable in the present invention is not particularly limited, but for example mention may be made of an organic pigment, inorganic pigment, metal oxide, metal hydroxide, mica, pearl agent, metal, magnetic powder, silicate ore, resin powder, powder having rubbery elasticity, or a porous substance alone or in combinations of two or more types.

[0010]

Particularly preferable among these are any inorganic powders having particle sizes of not more than 1 mm (sometimes including particles larger than 1 mm). Specifically, metal oxides, metal hydroxides, clay minerals, pearl agents, metals, carbon, magnetic powder, silicate ores, porous materials, etc. may be mentioned by way of illustration.

[0011]

These powders may be used alone or in combinations of several types. Further, they may be in a coagulated mass or in

the form of a molded article or shaped article. According to the present invention, it is possible to improve (treat) any inorganic powder including even superfine powder of a particle size of not more than 0.02 μm.

[0012]

Here, as specific examples of inorganic pigments (including metal oxides and metal hydroxides), Prussian Blue, Ultramarine, Mangan Violet, titanium (oxide) coated mica, magnesium oxide, aluminum oxide, aluminum hydroxide, silica, iron oxides (α-$FeeO_5$, γ-Fe_2O_3, Fe_2O_4, FeO, FeOOH, etc.), yellow iron oxide, black iron oxide, iron hydroxide, titanium oxide (in particular titanium dioxide with a particle size of 0.001 to 1 μm), lower titanium oxide, cerium oxide, zirconium oxide, chromium oxide, chromium hydroxide, manganese oxide, cobalt oxide, nickel oxide, etc. and composite oxides and composite hydroxides obtained by combinations of two or types of the same, for example, silica-alumina, iron titanate, cobalt titanate, lithium cobalt titanate, cobalt aluminate, etc. may be mentioned. In addition, as nonoxides, bismuth oxychloride, boronitride, silicon nitride, titanium nitride, and other nonoxide ceramic powders may be mentioned.

[0013]

The silicone-treated powder of the present invention has almost all of the residual Si-H groups cross-linked or substituted with inert functional groups and has no active functional groups adsorbed on the surface, so is a silicone-treated powder which is almost completely free of production of hydrogen, exhibits sufficient hydrophobicity, and is stable and good in quality.

The amount of hydrogen produced by the Si-H groups residually present on the surface of the silicone-treated powder of the present invention is not more than 0.2 ml/g of treated powder, more preferably not more than 0.1 ml/g of treated powder. If the amount of hydrogen produced exceeds 0.2 ml/g of treated powder, there is an accompanying risk at the

time of production of a cosmetic or the shelf life of the product is obstructed in some cases. Further, the contact angle of water with the treated powder is not less than 100°, more preferably 100 to 130°. If the contact angle of water is less than 100°, the functions and stability of the product are sometimes hindered.

[0014]

The silicone-treated powder of the present invention may be produced by the above process of production of a silicone-treated powder according to the present invention. As the silicone compound having an Si-H group in the silicone compounds usable for the process of production, one of the following general formula (1)

[0015]

[Chemical 2]

$$(R^1HSiO)_a(R^2R^3SiO)_b(R^4R^5R^6SiO_{1./2})_c \quad \ldots (1)$$

[0016]

[wherein, (R^1, R^2, and R^3 are mutually independently a hydrogen atom or a C_1 to C_{10} hydrocarbon group substitutable by at least one halogen atom (provided that R^1, R^2, and R^3 are never simultaneously hydrogen atoms), R^4, R^5, and R^6 are mutually independently a hydrogen atom or a C_1 to C_{10} hydrocarbon group substitutable by at least one halogen atom, a is an integer of 1 or more, b is 0 or an integer of 1 or more, c is 0 or 2 (provided that $3 \leq a+b+c \leq 10000$), and the compound includes at least one Si-H group portion] is preferable, more preferably it is methylhydrogenpolysiloxane, a methylhydrogenpolysiloxane-dimethylpolysiloxane copolymer, or tetramethylcyclotetrasiloxane.

[0017]

As silicone compounds other than silicone compounds having an Si-H group usable in the process of the present invention, there are for example dimethylpolysiloxane, octamethylcyclotetrasiloxane, etc.

[0018]

The amount of the silicone compound with respect to the

powder usable in the process of the present invention is 0.1 to 20.0 wt%, preferably 0.5 to 15.0 wt%.

[0019]

In the process of production of a silicone-treated powder of the present invention, in the first step, that is, the silicone treatment step, the silicone compound is brought into contact with the above various powders in the form of a vapor of itself, in the form of a solution dissolved in a suitable solvent, or in the form of a liquid of itself.

When bringing the silicone compound into contact with the powder in the form of a vapor, for example, a cyclic organosiloxane and powder are placed in separate containers in a sealed space and the tops left open or a treatment agent is mixed with a carrier gas and introduced into a chamber loaded with the powder, so no special hardware is required.

When bringing the silicone compound into direct contact with the powder in the form of a liquid, use is made of a suitable mixer, for example, a rotary ball mixer, a vibration type ball mixer, a planetary type ball mixer, a sand mill, an attritor, a bag mill, a pony mixer, a planetary mixer, an agitator, a Henschel mixer, etc.

When bringing the silicone compound into contact with the powder in a solution, for example, a solution containing 0.3 to 50 wt% of the compound in a solvent such as alcohol, water, hexane, cyclohexane, and toluene is prepared, the powder made to disperse in it, then the solution heated to evaporate the solvent and the silicone compound made to polymerize on the surface. This may be done using a Henschel mixer, a kneader, a mill using beads, etc.

[0020]

In the process of production of a silicone-treated powder of the present invention, in the second step of heat treating the powder with which the silicone compound is mixed, the heating temperature and time of the powder is 350 to 500°C for 0.1 to 24 hours, preferably 1 to 4 hours, preferably 350 to 450°C for 1 to 4 hours. If less than 350°C, the Si-H groups do

not easily react, while if over 500°C, the burning and decomposition of the $Si-CH_3$ groups are promoted, and the hydrophobicity declines or disappears (hydrophilicity), that is, the silicone is converted to silica.

[0021]

Further, as the heating atmosphere, it is possible to heat the powder in the air, which is an atmosphere containing moisture, or in another gas containing moisture of an extent of the moisture in the air. In addition, it is possible to adjust the powder in an atmosphere not containing moisture, then heat while adding moisture during the treatment (heating). As the device used for heating, an electric furnace, tunnel furnace, rotary kiln, etc. may be used.

[0022]

According to the present invention, further, there are provided a cosmetic composition, paint composition, and resin shaped article (container etc. formed by injection molding). In the processes of production of these, it is possible to produce products by ordinary methods other than the use of the silicone-treated powder according to the present invention instead of powder treated by a conventional method. The cosmetic composition, paint composition, and resin shaped article obtained in the present invention enable a reduction of the costs of manufacture of the products, an improvement of the quality of the products, stability of the products, and a reduction in the load in work.

[0023]

[Examples]

Next, the present invention will be explained in further detail according to the Examples. The scope of the present invention is not however limited by these Examples in any way. The units of amounts blended are wt%.

[0024]

Example 1-1

500 g of sericite (average particle size: 4 μm) and 15 g of methylhydrogenpolysiloxane (product name: Silicone KF99,

made by Shin-Etsu Chemical) were dissolved in 50 ml of hexane. This solution was placed in a Henschel mixer and stirred and mixed at room temperature for a predetermined time, then was placed in a dryer of 100°C to evaporate the solvent. Next, the powder was placed in an electric furnace set to 400°C in advance and heated for 3 hours to obtain a silicone-treated powder.

[0025]

Example 1-2

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to titanium dioxide.

[0026]

Example 1-3

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to silica.

[0027]

Example 1-4

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to talc.

[0028]

Example 1-5

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to zinc white.

[0029]

Example 1-6

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to titanated mica.

[0030]

Example 1-7

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1

to bengara.

[0031]

Example 1-8

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to yellow iron oxide.

[0032]

Example 1-9

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to black iron oxide.

[0033]

Example 1-10

The same procedure was followed to obtain a silicone-treated powder except for changing the sericite of Example 1-1 to mica.

[0034]

Example 2-1

500 g of finely divided titanium dioxide particle and 35 g of tetramethylcyclotetrasiloxane were placed in a desiccator and allowed to stand at 50°C for one day, then the powder was heated by passing it through a tunnel furnace set to 300°C in advance (nitrogen atmosphere containing moisture) over 10 minutes to obtain a silicone-treated powder.

[0035]

Example 2-2

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to finely divided particle zinc oxide.

[0036]

Example 2-3

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to bengara.

[0037]

Example 2-4

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to black iron oxide.

[0038]

Example 2-5

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 t carbon black.

[0039]

Example 2-6

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to titanium mica.

[0040]

Example 2-7

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to a titanium dioxide/iron oxide composite.

[0041]

Example 2-8

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to chromium oxide.

[0042]

Example 2-9

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to Ultramarine.

[0043]

Example 2-10

The same procedure was followed to obtain a silicone-treated powder except for changing the finely divided particle titanium dioxide of Example 2-1 to finely divided particle cerium dioxide.

[0044]

Example 3-1

The method of Example 1-1 was used to coat 500 g of sericite with silicone, then this was placed in an electric furnace set to a dry nitrogen atmosphere and raised in temperature. After reaching 400°C, 10 g of water was dropped from above at a rate of 1/6 g/min. After dropping was finished, the powder was further heated for 1 hour to obtain a silicone-treated powder.

[0045]

Comparative Examples 1-1 to 1-10

The same procedures were followed by the same powders and the same methods as the corresponding Examples 1 to obtain silicone-treated powders except for not performing the heating step.

[0046]

Comparative Examples 2-1 to 2-10

The same procedures were followed by the same powders and the same methods as the corresponding Examples 1 to obtain silicone-treated powders except for performing the heating step at 300°C for 4 hours.

[0047]

Comparative Examples 3-1 to 3-10

The same procedures were followed by the same powders and the same methods as the corresponding Examples 1 to obtain silicone-treated powders except for performing the heating step at 550°C for 4 hours.

[0048]

The amounts of production of hydrogen gas of the silicone-treated powders obtained in the Examples and the Comparative Examples and their contact angles with water were measured by the following methods:

The amount of production of hydrogen gas was measured by the gas burette method. 2 g of silicone-treated powder and about 40 ml of alcohol were placed in a three-necked flask. About 1 ml of 10% NaOH aqueous solution was dropped in this by

a closed system to cause the production of hydrogen gas and the amount of production of hydrogen (ml) per g was calculated.

The contact angle with water was measured by using an IR tableting machine (diameter 13 mm) to prepare pellets of the silicone-treated powders of the Examples and Comparative Examples and then using an automatic contact angle meter (Model CA-Z) made by Kyowa Kaimen Kagaku (average value for three measurements).

[0049]

The results of the measurement of the amounts of hydrogen production and the contact angles with water in the silicone-treated powders obtained in the Examples and the Comparative Examples are shown in Tables 1 to 4. The smaller the amount of residual Si-H groups acting as the source of the production of hydrogen gas and the higher the contact angle, the better.
the contact angle, the better.

[0050]

[Table 1]

Example	Amount of production of residual hydrogen (ml/g)	Contact angle
Ex. 1-1	0.08	115
Ex. 1-2	0.01	120
Ex. 1-3	0.10	107
Ex. 1-4	0.03	109
Ex. 1-5	0.02	115
Ex. 1-6	0.01	116
Ex. 1-7	0.04	122
Ex. 1-8	0.05	117
Ex. 1-9	0.03	107
Ex. 1-10	0.12	115
Ex. 2-1	0.11	120
Ex. 2-2	0.06	110
Ex. 2-3	0.08	115
Ex. 2-4	0.05	107
Ex. 2-5	0.03	124
Ex. 2-6	0.13	121
Ex. 2-7	0.08	113
Ex. 2-8	0.09	110
Ex. 2-9	0.05	119
Ex. 2-10	0.0	125
Ex. 3-1	0.02	120

[0051]

[Table 2]

Comp. example	Amount of production of residual hydrogen (ml/g)	Contact angle
Comp. Ex. 1-1	2.81	120
Comp. Ex. 1-2	1.35	122
Comp. Ex. 1-3	2.45	109
Comp. Ex. 1-4	2.08	110
Comp. Ex. 1-5	1.55	115
Comp. Ex. 1-6	1.87	117
Comp. Ex. 1-7	2.14	120
Comp. Ex. 1-8	2.00	114
Comp. Ex. 1-9	2.33	105
Comp. Ex. 1-10	2.52	115

[0052]

[Table 3]

Comp. example	Amount of production of residual hydrogen (ml/g)	Contact angle
Comp. Ex. 2-1	1.88	117
Comp. Ex. 2-2	0.98	125
Comp. Ex. 2-3	1.65	103
Comp. Ex. 2-4	1.22	114
Comp. Ex. 2-5	1.04	107
Comp. Ex. 2-6	1.13	124
Comp. Ex. 2-7	1.64	127
Comp. Ex. 2-8	1.33	110
Comp. Ex. 2-9	1.79	104
Comp. Ex. 2-10	1.50	113

[0053]

[Table 3]

Comp. example	Amount of production of residual hydrogen (ml/g)	Contact angle
Comp. Ex. 3-1	0.0	0
Comp. Ex. 3-2	0.0	0
Comp. Ex. 3-3	0.0	0
Comp. Ex. 3-4	0.0	0
Comp. Ex. 3-5	0.0	0
Comp. Ex. 3-6	0.0	0
Comp. Ex. 3-7	0.0	0
Comp. Ex. 3-8	0.0	0
Comp. Ex. 3-9	0.0	0
Comp. Ex. 3-10	0.0	0

[0054]

As a result of the above measurement, there is still a certain amount of hydrogen generation (i.e., Si-H group is remained) at a heating temperature of 300°C after the silicone treatment and the hydrogen generation was stopped at 550°C with loss of the hydrophobicity. On the other hand, in the case of the heating at 400°C, the hydrogen generation is substantially stopped but good hydrophobicity is maintained.

[0055]

Example 4 Foundation

	Ingredient	wt%
(1)	Treated powder of Example 1-1	35.0
(2)	Treated powder of Example 1-2	13.0
(3)	Treated powder of Example 1-4	24.7
(4)	Treated powder of Example 1-10	10.0
(5)	Treated powder of Example 1-7	1.0
(6)	Treated powder of Example 1-8	2.5
(7)	Treated powder of Example 1-9	0.1
(8)	Liquid paraffin	8.0
(9)	Sorbitan sesquioleate	3.5
(10)	Glycerin	2.0
(11)	Ethyl paraben	0.2

(Process of Production)

The ingredients (1) to (7) were mixed and pulverized by a

pulverizer. The result was transferred to a high speed blender, then the ingredient (10) was added and the result mixed. Separate from this, the ingredients (8), (9), and (11) were mixed to make them homogeneous, then this was added to the above mixture and further mixed to make them homogeneous. The mixture was then treated by a pulverizer and passed through a sieve to obtain a standard particle size, then the result was compression molded to obtain a solid foundation.

[0056]

Comparative Example 4

The same procedure was followed as in Example 4 to prepare a foundation except for replacing ingredients (1) to (7) in the foundation prepared in Example 4 with the ingredients of the corresponding Comparative Example 1.

[0057]

Comparative Example 5

The same procedure was followed as in Example 4 to prepare a foundation except for replacing the ingredients (1) to (7) in the foundation prepared in Example 4 with the ingredients of the corresponding Comparative Example 2.

[0058]

Comparative Example 6

The same procedure was followed as in Example 4 to prepare a foundation except for replacing the ingredients (1) to (7) in the foundation prepared in Example 4 with the ingredients of the corresponding Comparative Example 3.

[0059]

(1) Evaluation of Use

Samples held at 50°C for one month were evaluated by the following criteria as for various aspects of usability (removability, covering power, slip, use by a sponge wet with water, cracking of the pack surface, hold, transparency, and water resistance) by a panel of 20 women:

(Evaluation Criteria)

Very good: At least 17 women responded sample was good

Good: 12 to 16 women responded sample was good

Fair: 9 to 11 women responded sample was good

Poor: 5 to 8 women responded sample was good

Very poor: 4 or less women responded sample was good

[0060]

(2) Evaluation of Shelf Life

Samples held at 50°C for one month were compared for stability.

[0061]

(3) Evaluation of SPF (UV Blocking Effect)

Samples held at 50°C for one month were measured for in vitro SPF value by the Spectro Radiometer method.

[0062]

The results of evaluation of the usability of the samples of Example 4 and Comparative Examples 4 to 6 by the above criteria after being held at 50°C for one month are shown in Table 5.

[0063]

[Table 5]

	Ex. 4	Comp. Ex. 4	Comp. Ex. 5	Comp. Ex. 6
Removability	Very good	Fair	Fair	Fair
Covering power	Very good	Fair	Fair	Fair
Slip	Good	Good	Good	Poor
Use on sponge wet with water	No problem	No problem	No problem	Caking
Cracking of pack surface	None	Yes	Yes	None

[0064]

As will be understood from Table 5, Example 4 could be applied with no problem even using water as a dual use type and was superior in shelf life.

[0065]

Example 5 Emulsion Foundation

	Ingredient	wt%
(A)	Ion exchanged water	43.5
	Sodium chondroitin sulfate	1.0
	1,3-butylene glycol	3.0
	Methyl paraben	q.s.

(B)	Dimethylpolysiloxane (20 cs)	16.0
	Decamethylcyclopentasiloxane	5.0
	Silicone resin	1.0
	Polyoxyalkylene modified organopolysiloxane (modification rate 20%)	4.0
	Antioxidant	q.s.
	Fragrance	q.s
(C)	Treated powder of Example 1-8	1.0
	Treated powder of Example 2-3	0.45
	Treated powder of Example 2-4	0.2
	Treated powder of Example 1-2	11.7
	Treated powder of Example 1-1	9.65
	Treated powder of Example 2-7	2.0

(Process of Production)

The ingredients (B) were heated to melt, then the powders of ingredient (C) were added and dispersed in them. Further, the ingredients (A) melted and heated in advance were added to make an emulsion, then the emulsion was cooled to room temperature to obtain an emulsion foundation.

[0066]

Comparative Example 7

The same procedure was followed as in Example 5 to obtain an emulsion foundation except for replacing the ingredients (C) in the emulsion foundation prepared in Example 5 with the ingredients of the corresponding Comparative Example 1.

[0067]

Comparative Example 8

The same procedure was followed as in Example 5 to obtain an emulsion foundation except for replacing the ingredients (C) in the emulsion foundation prepared in Example 5 with the ingredients of the corresponding Comparative Example 2.

[0068]

Comparative Example 9

The same procedure was followed as in Example 5 to obtain an emulsion foundation except for replacing the ingredients (C) in the emulsion foundation prepared in Example 5 with the

ingredients of the corresponding Comparative Example 3.

[0069]

The results of evaluation of the usability of the samples and shelf life of Example 5 and Comparative Examples 7 to 9 by the above criteria after being held at 50°C for one month are shown in Table 6.

[0070]

[Table 6]

	Ex. 5	Comp. Ex. 7	Comp. Ex. 8	Comp. Ex. 9
Covering power	Good	Fair	Fair	Fair
Slip	Good	Fair	Fair	Poor
Hold	Very good	Good	Good	Very poor
Shelf life	No problem	Container swelled	Container swelled	No problem

[0071]

As will be understood from Table 6, the emulsion foundation prepared in Example 5 had a good hold and was superior in shelf life as well.

[0072]

Example 6 Pressed Powder

	Ingredient	wt%
(1)	Treated powder of Example 1-5	30.0
(2)	Treated powder of Example 1-4	65.8
(3)	Iron oxide pigment	0.1
(4)	Squalane	2.0
(5)	2-ethylhexyl palmitate	2.0
(6)	Fragrance	0.1

(Process of Production)

The ingredients (1), (2), and (3) were mixed in a Henschel mixer, then a heated mixture of the ingredients (4) and (5) was sprayed on the mixture. These were mixed, then pulverized, then molded into a dish obtain a pressed powder. The obtained pressed powder had a moisture retention effect, a good hold, and superior shelf life as well.

[0073]

Example 7 Body Powder

Ingredients wt%

(A)	Treated powder of Example 1-4	89.0
	Treated powder of Example 1-6	10.0
	Coloring pigment	q.s.
(B)	Treated powder of Example 1-5	3.0
(C)	Magnesium stearate	4.0
	Liquid paraffin	1.0
	Bactericide	q.s.
(D)	Fragrance	q.s.

(Process of Production)

The ingredients (A) were mixed by a blender, then the ingredient (B) was added and mixed well. The ingredients (C) were then added, the coloring adjusted, then the ingredient (D) was sprayed on and then homogeneously mixed in. The mixture was pulverized by a pulverizer, then passed through a sieve to obtain the body powder. The obtained body powder had a high water repellency.

[0074]

Example 8 Lipstick

	Ingredient	wt%
(1)	Hydrocarbon wax	3.0
(2)	Carnauba wax	1.0
(3)	Glyceryl isostearate	40.0
(4)	Liquid paraffin	45.8
(5)	Treated powder of Example 1-3	4.0
(6)	Mixed treated powders of Example 1-1 and Example 1-7	6.0
(7)	Fragrance	0.2

(Process of Production)

The ingredients (1) to (4) were melted at 85°C, then the ingredients (5) and (6) were added while stirring. Next, while stirring, the ingredient (7) was added and the mixture packed into a container. The obtained lipstick was superior in moisture retention effect.

[0075]

Example 9 Water-in-Oil Type Emulsion Sunscreen

Ingredient	wt%

(A)	Decamethylcyclopentasiloxane	Bal.
	Dimethylpolysiloxane	5.0
	Polyoxyethylene-methylpolysiloxane copolymer	3.0
	Organic modified bentonite	1.0
(B)	Treated powder of Example 1-4	10.0
	Treated powder of Example 2-1	7.0
	Treated powder of Example 2-2	10.0
	Silicone elastic powder	3.0
	Fragrance	q.s.
	Antioxidant	q.s.
(C)	Ion exchanged water	35.0
	Glycerin	5.0
	Preservative	q.s.

(Process of Production)

The (A) phase was heated to melt, then the (B) phase was added and the mixture was homogeneously dispersed by a homomixer. Then phase (C) was added gradually and stirred well, then homogeneously emulsified by a homomixer. This was then stirred and cooled to obtain a water-in-oil type emulsion sunscreen. The obtained sunscreen had a high sunburn preventing effect.

[0076]

Comparative Example 10

The same procedure was performed as in Example 9 to prepare a water-in-oil type emulsion sunscreen except for replacing the treated powder portion in the ingredients (B) in the water-in-oil type emulsion sunscreen prepared in Example 9 with the ingredients of the corresponding Comparative Example 1.

[0077]

Comparative Example 11

The same procedure was performed as in Example 9 to prepare a water-in-oil type emulsion sunscreen except for replacing the treated powder portion in the ingredients (B) in the water-in-oil type emulsion sunscreen prepared in Example 9

with the ingredients of the corresponding Comparative Example 2.

[0078]

Comparative Example 12

The same procedure was performed as in Example 9 to prepare a water-in-oil type emulsion sunscreen except for replacing the treated powder portion in the ingredients (B) in the water-in-oil type emulsion sunscreen prepared in Example 9 with the ingredients of the corresponding Comparative Example 3.

[0079]

The results of evaluation of the usability, the SPF value, and the shelf life of the samples of Example 9 and Comparative Examples 10 to 12 by the above criteria after being held at 50°C for one month are shown in Table 7.

[0080]

[Table 7]

	Ex. 9	Comp. Ex. 10	Comp. Ex. 11	Comp. Ex. 12
Slip	Good	Fair	Fair	Poor
Transparency	Very good	Good	Good	Fair
Water resistance	Very good	Good	Good	Very poor
SPF	44	41	42	22
Shelf life	No problem	Container swelled	Container swelled	No problem

[0081]

Comparative Example 10 and Comparative Example 11 were good in usability to a certain extent, but the containers swelled along with time, Comparative Example 22 suffered from aggregation of the powder due to the hydrophilicity and had a low SPF, but Example 9 was superior in all of the usability, shelf life, and SPF value.

[0082]

Example 10 Paint

20 g of the treated powder obtained in Example 1-2 and 18 g of acrylic resin solution (Mn=48,200, Mn/Mw=2.56) were mixed together with 70 g of glass beads by a paint shaker for 20

minutes to obtain a paint. The obtained paint was superior in paint stability over time.

[0083]

Example 11 Container

The treated powder obtained in Example 2-1 was mixed in an amount of 2 wt% in polyethylene. This was then injected molded into a white polystyrene wide mouth vase.

[0084]

Comparative Example 13

As a comparative example, the same procedure was used as in Example 11 for injection molding except for using finely divided particle titanium dioxide not treated with silicone.

[0085]

4 cm x 4 cm sized pieces were cut from the wide mouth vases of Example 11 and Comparative Example 13 and measured for UV absorption spectra (diffusion reflection method), whereupon the piece obtained from Example 11 was observed to have a higher UV absorption effect.

[0086]

[Effects of the Invention]

As explained in detail above, the silicone-treated powder of the present invention was stable in quality and free from any unpleasant odor from the powder. Further, the silicone-treated powder of the present invention can be used for cosmetic compositions, paints, resin shaped articles, and a broad range of other products.

Further, according to the process of production of a silicone-treated powder of the present invention, there are the advantages that it is possible to produce a good quality silicone-treated powder by a simple process and possible to provide it at a low production cost.

[Name of Document] Abstract

[Abstract]

[Problem] To provide a silicone-treated powder capable of formulating into various cosmetics and having an excellent stability in the formulated product and a production process thereof.

[Means for Solution] A silicone-treated powder comprised of a powder coated on its surface with a silicone compound, wherein an amount of hydrogen produced by Si-H groups residually present on the surface of the silicone-treated powder is not more than 0.2 ml/g of treated powder and a contact angle of water with the treated powder is at least 100°. In addition, in the production process thereof, the silicone compound coated powder having a silicone compound coated on the surface of the powder is heated at 350 to 500°C for 0.1 to 24 hours.

[Selected Drawing] None